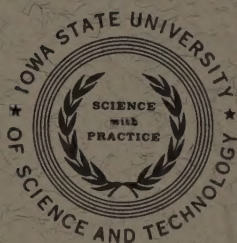


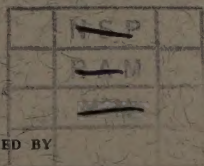
IOWA STATE JOURNAL OF SCIENCE

A Quarterly of Research

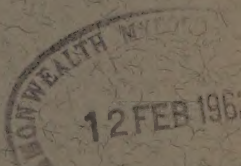


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PUBLISHED BY



The Iowa State University Press, Ames, Iowa, U.S.A.

IOWA STATE
JOURNAL OF SCIENCE

A Quarterly of Research

Published August, November, February, and May

EDITOR-IN-CHIEF R.E. BUCHANAN
BUSINESS MANAGER MARSHALL TOWNSEND

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All matters pertaining to subscriptions, remittances, etc., should be
addressed to the Iowa State University Press, Press Building, Ames,
Iowa. Subscriptions are as follow: Annual \$6.00 (Canada, \$6.50, other
foreign countries \$7.00); single copies \$2.00.

Entered as second-class matter January 16, 1935, at the post office at
Ames, Iowa, under the Act of March 3, 1879.

EVALUATING THE EFFECT OF EUROPEAN CORN BORER
POPULATIONS ON CORN YIELD^{1,2}

J.L. Jarvis³, T.R. Everett³, T.A. Brindley^{3,4}, and F.F. Dicke³

ABSTRACT. First-brood infestation of a borer-susceptible, single-cross dent corn by the European corn borer (*Pyrausta nubilalis* (Hbn.)) resulted in a greater yield reduction than did infestation by the second brood. Both cavities in the stalk and lesions in the leaves were a better index of damage than were larvae. A greater yield loss per unit of damage occurred in the late planting than in the early planting. A 2.3% yield reduction per cavity occurred in the early planting in 1958 and a 1% reduction in 1959. In the late planting in 1958 a 4.1% yield loss per cavity occurred. A 1.4% reduction per leaf lesion occurred in the early planting in 1958 and a 1.7% reduction in the late planting. In the fall, cavities in both split and dissected stalks gave a better estimate of yield reduction than did larvae. Cavity counts in split stalks were as reliable in estimating damage as cavity counts in dissected stalks. Because splitting requires about one-fourth the time required to dissect a stalk, this method is proposed for determining borer damage. The yield reduction per cavity in the fall in both 1958 and 1959 averaged between 1 and 2%. When yield loss occurs, the reduction is caused by infestation either by the first or the second brood, or by both, without an additional effect owing to the interaction of infestation by both broods.

- - - - -

Several methods have been used in the past for evaluating damage done to field corn by the European corn borer (*Pyrausta nubilalis* (Hbn.)). Early attempts at assaying losses were based on the amount of stalk breakage in a field and farmer estimates of yields. Patch *et al.* (1938), working with the univoltine strain of the corn borer, used the number of larvae per plant as an index of yield loss. Everett *et al.* (1958) and Kwolek and Brindley (1959), working with the bivoltine strain, showed that the number of cavities in the stalk was a more reliable index of yield

¹ Accepted for publication May 26, 1961. Journal paper No. J-4019 of the Iowa Agricultural and Home Economics Experiment Station, Ames, Iowa. Project No. 1193.

² Contribution from North Central Regional Project NC-20.

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loss than the number of larvae. Everett *et al.* (1958) also demonstrated an inverse relationship between leaf lesions resulting from first-brood infestation and yield, but concluded that the best index of yield loss was the number of cavities or larvae per plant at the end of first-brood infestation. As the result of findings by Patch *et al.* (1942), a standard estimate of "3% loss per borer per plant," based on numbers of larvae in the fall, has been used to determine the yield reduction due to borer infestation.

The purpose of this paper is to evaluate damage done by the European corn borer to dent corn, and to compare the reliability of several indices of plant infestation for estimating yield loss.

MATERIALS AND METHODS

The data given in this paper were obtained in 1958 and 1959 from experiments conducted at Ankeny, Iowa, to study the effect of corn borer infestation on yield. The experimental design used was a split-plot with five replications. The plots were planted with two corn hybrids (borer resistant Oh43 x Oh51A and borer susceptible WF9 x M14) and two plantings (early and late) of each were made. These plantings were designated as whole-plot treatments. The sub-plot treatments consisted of a factorial arrangement of all possible combinations of three levels of first-brood and three levels of second-brood infestation, as follows:

1. The plants were sprayed with EPN at the rate of 1/2 pound of toxicant per acre beginning at first egg hatch and repeated at 5-day intervals for as long as it was required to control first-brood larvae. Plants were sprayed with DDT at the rate of 1 pound of actual toxicant per acre to control second-brood larvae.

2. The plants were sprayed with EPN to control first-brood larvae and were naturally infested by second-brood larvae.

3. The plants were sprayed with EPN to control first-brood larvae. For the second-brood treatment, these plants received a natural infestation plus an artificial infestation of three second-brood egg masses per plant. These egg masses were applied in the vicinity of the ear to simulate natural oviposition. The egg masses were produced in the laboratory from field-collected moths.

4. The plants were naturally infested by first-brood larvae and sprayed with DDT to control second-brood larvae.

5. The plants were naturally infested by both first- and second-brood larvae.

6. The plants were naturally infested by first-brood larvae. For the second-brood treatment, these plants received a natural infestation plus an artificial infestation of three second-brood egg masses per plant.

7. These plants received a natural infestation of first-brood larvae plus an artificial infestation of three egg masses per plant, which were dropped into the whorl. The plants were sprayed with DDT to eliminate second-brood infestation.

8. These plants were naturally infested by first-brood larvae with an application of three egg masses per plant added, and were naturally infested by second-brood larvae.

9. These plants received a natural first-brood infestation plus three egg masses per plant. The second-brood treatment was natural infestation plus three second-brood egg masses.

These treatments were planned to provide different intensities of both first- and second-brood borer infestation.

Each plot consisted of a block of 6 x 7 hills. Yields were taken from a 2- x 5-hill area in the center of each plot; hills used to determine yields consisted of three plants and were bordered on all sides by hills which contained at least two plants. First-brood infestation was determined by the dissection of six plants taken at random from either side of the yield hills, with no more than one plant being taken from each hill.

Three indices were used to evaluate the yield loss due to the effect of first-brood infestation: (1) the numbers of leaf lesions per plant, (2) the numbers of larvae per plant, and (3) the numbers of cavities per plant. These indices were determined by dissecting plants from treatments 1, 4, and 7 at the end of the period of first-brood infestation. These three treatments represent three levels of first-brood infestation (none, natural, and natural plus three egg masses), and were kept as free as possible from second-brood infestation by spraying the plants with DDT. Therefore any reduction in yield with treatments 4 and 7 may be attributed to damage by first-brood larvae.

The effect of second-brood infestation, and infestation by both broods, was evaluated by three indices. One plant from each hill of the 2 x 5 yield plots was dissected to determine the number of larvae and cavities present. A second plant from each hill was split longitudinally and the cavities counted in the stalk above and below the primary ear node and in the ear shank. No larval counts were made from split stalks. The effect of second-brood infestation only was determined from treatments 1, 2, and 3. These treatments were kept as free as possible from first-brood infestation and received three levels of second-brood infestation. Thus any yield differences within these treatments was attributed to second-brood infestation.

All ears of corn from the designated 2- x 5-hill portion of each plot were harvested and weighed. Yield, in bushels per acre, was computed on the basis of 15.5% moisture.

RESULTS

Effect of First Brood on Yield

Data collected from stalks infested with the first-brood European corn borer and yields from these same plots are summarized in Table 1.

In order to evaluate the three indices of first-brood borer damage, correlation coefficients were computed from data on yield in bushels per acre and the average number of leaf lesions, larvae, or cavities per plant (Table 2). When the correlation coefficient was significant, a regression equation was computed (Table 3).

The results indicated that a straight line fits the data within the range of values found in the data presented. However, extrapolation beyond the limits of the data should not be attempted because of possible curvilinearity of the regression lines beyond this point.

Table 1. Data collected from dent corn stalks infested with first-brood European corn borer, 1958-59. Average of five replications.

Year	Hybrid Planting ^a	Treatment No.	Per plant			Yield (bu/A)
			Leaf lesions	Larvae	Cavities	
1958	ES	1	0.43	0.03	0.03	128.6
		4	5.10	2.23	3.20	123.2
		7	8.83	4.00	5.83	110.9
	ER	1	0.63	0.13	0.17	103.5
		4	3.13	1.03	1.73	101.7
		7	4.73	1.80	2.57	97.9
	LS	1	0.20	0	0.03	120.6
		4	0.23	0.13	0.13	116.9
		7	7.00	2.90	3.30	104.0
	LR	1	0.07	0.03	0.03	99.7
		4	0.30	0.07	0	103.2
		7	1.17	0.63	0.63	100.8
1959	ES	1	0.17	0	0	110.6
		4	0.67	0.20	0.37	112.7
		7	2.13	1.00	2.00	106.6
	ER	1	0.17	0	0.03	99.8
		4	0.63	0.13	0.30	98.4
		7	1.30	0.17	0.70	98.8
	LS	1	0.07	0	0	107.6
		4	0.20	0	0	109.3
		7	1.07	0.13	0.27	103.5
	LR	1	0	0	0	96.3
		4	0.10	0	0	97.5
		7	0.20	0.07	0.17	95.8

^a E = Early, L = Late, R = Resistant, S = Susceptible.

Analysis of the 1958 data showed significant correlations between yield and leaf lesions, larvae, or cavities in both the early and late planted susceptible hybrid. In 1959, only the correlation between cavities and yield in the early susceptible hybrid was significant. Natural infestation, survival, and damage were considerably less in 1959 than in 1958. The limited range of infestation values and variation in yield in 1959 depreciated the correlation between borer injury and yield.

The regression equations are given in Table 3 to show the amount of yield loss from each unit of borer damage. The regression lines for these equations are shown in Figures 1, 2, and 3. Yield reduction per

Table 2. Correlation coefficients of dent corn yield with leaf lesions, larvae, and cavities at end of first-brood infestation of European corn borer, 1958-59.

Year	Hybrid planting ^a	Leaf lesions and yield	Larvae and yield	Cavities and yield
1958	ES	-0.734**	-0.886**	-0.836**
	ER	-0.431	-0.462	-0.445
	LS	-0.864**	-0.874**	-0.869**
	LR	0.161	-0.148	-0.101
1959	ES	-0.372	-0.489	-0.601*
	ER	-0.123	-0.149	-0.089
	LS	-0.345	-0.481	-0.434
	LR	-0.089	0.325	0.282

^a E = Early, L = Late, R = Resistant, S = Susceptible.

* Significant at the 5% level.

** Significant at the 1% level.

Table 3. Regression equations relating yield of dent corn in bushels per acre and yield loss to leaf lesions, larvae, and cavities per plant after stalk dissection at end of first-brood infestation by European corn borer, 1958-59.

Year	Hybrid planting ^a	Regression equation	Standard error of regression coefficient	Yield loss per unit of damage ^b (%)
<u>Leaf lesions and yield</u>				
1958	ES	$Y = 129.3 - 1.76X$	0.45	1.4
	LS	$Y = 119.0 - 2.09X$	0.34	1.7
<u>Larvae and yield</u>				
1958	ES	$Y = 131.4 - 5.05X$	0.74	3.8
	LS	$Y = 119.1 - 5.16X$	0.80	4.3
<u>Cavities and yield</u>				
1958	ES	$Y = 130.0 - 3.03X$	0.55	2.3
	LS	$Y = 119.1 - 4.51X$	0.66	4.1
1959	ES	$Y = 110.8 - 1.10X$	0.69	1.0

^a E = Early, L = Late, R = Resistant, S = Susceptible.

^b Leaf lesions, larvae, or cavities.

larva was greater than the unit reduction for either leaf lesions or cavities. The greater yield reduction associated with larvae was owing to the fact that, although a larva had produced damage, it was not necessarily recovered. Some cavities did not contain larvae because of mortality due to predation or disease, or because of larval migration to other plants. The larger standard errors obtained when the numbers of larvae were used as the independent variable indicate that this is not as reliable an index to estimate borer damage as either leaf lesions or cavities. The loss in yield per cavity was somewhat greater than the loss per leaf lesion although the standard errors when leaf lesions were used were lower than when cavities were used. When a significant yield reduction, owing to first-brood borer infestation, occurred in both the early and late plantings, the reduction per unit of damage was greater in the late planting. Statistically this difference was not significant, but the consistently greater yield reduction in the late planting indicates that a first-brood infestation in late planted corn caused more yield loss per unit of damage than an infestation of early corn. However, it should be pointed out that in both 1958 and 1959 natural infestation in the late plantings was quite low and that yield reduction due to first-brood borer infestation was the result of artificial and not natural infestation. Although delayed planting will reduce the amount of natural infestation, yield reduction in late planted corn will occur if a first-brood infestation is present.

The percent loss per unit of damage was computed. These figures are shown in Table 3. The yield reduction in the susceptible hybrid in 1958 was greater than in 1959. The percent loss was somewhat greater in the late than in the early susceptible hybrid. No significant yield reduction occurred in either the early or late planting of the resistant hybrid. Because the yield loss per unit of damage varies with the hybrid and date of planting, it is not possible to estimate with any degree of accuracy damage done by larvae of the first brood without knowledge of the corn hybrid, its degree of susceptibility, and the maturity of the corn plants at the time of infestation.

Yield reduction owing to borer infestation cannot be attributed to a single variable but is the result of a complex of factors. Since the three levels of first- and second-brood infestation were in a factorial arrangement, it was possible to detect yield differences owing to first-brood infestation, second-brood infestation, and the interaction of infestation by both broods.

The analysis of variance for the yield data is presented in Table 7. First-brood infestation in the late susceptible planting in 1959 resulted in a yield reduction, yet the correlation coefficients between yield and the indices of first-brood damage were not significant. Here is an example of reduced yield owing to borer infestation, yet loss as measured by leaf lesions, larvae, or cavities could not be demonstrated. This example clearly shows that first-brood corn borer infestation may result in a loss in yield even though the physical evidence of infestation may be slight.

Fall Dissection

All plots were dissected at the end of the growing season in order to determine the intensity of second-brood infestations, as well as the total

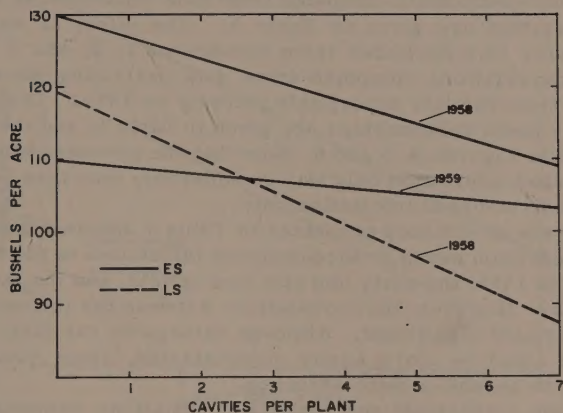


Figure 1. Regression lines for first-brood infestation relating cavities to yield, 1958-59.

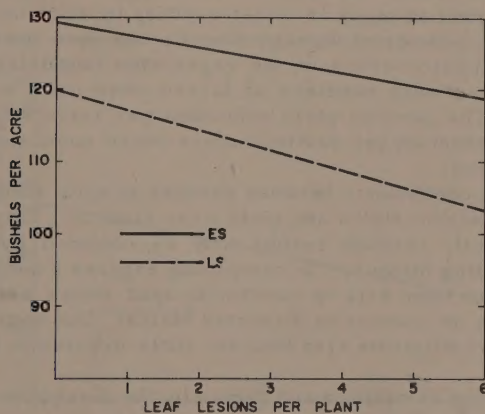


Figure 2. Regression lines for first-brood infestation relating leaf lesions to yield, 1958.

seasonal infestation on yield. Numbers of larvae per plant, cavities in split stalks, and cavities in dissected stalks were used as indices to relate damage done by the corn borer to yields. These data are given in Table 4.

Correlation coefficients computed from data collected at the time of the fall dissection are given in Table 5. The effect of second-brood infestation only was evaluated from treatments 1, 2, and 3. The only significant correlations computed from data pertaining to these treatments were from the late susceptible planting in 1959. The regression equations for these relationships are given in Table 6, and the regression lines plotted in Figures 4, 5 and 6. Note that the percent reduction owing to second-brood infestation only was considerably less than occurred as the result of first-brood infestation only.

The analysis of variance presented in Table 7 demonstrates a significant yield reduction owing to second-brood infestation in the late susceptible hybrid in 1958, the early and late susceptible, and the late resistant hybrid in 1959. However, the correlations between the indices of damage and yield were not significant. Although damage by the first brood was greater and could be more easily demonstrated, some yield loss did occur owing to second-brood infestation.

Correlation coefficients and regression equations computed from all treatments are given in Table 5 and 6. The regression lines are plotted in Figures 7, 8, and 9. This combination of the data is a composite of first-brood infestation only, second-brood infestation only, and different combinations of the two. Such a grouping might be expected in a random sample of fields from several farms.

The correlations between larvae and yield were consistently lower than correlations between either cavities in split stalks or cavities in dissected stalks and yield. These lower correlations would indicate that larvae are not as good an index as either cavities in split or cavities in dissected stalks in estimating damage done by the corn borer. As further proof, the standard errors of the regression coefficients given in Table 6 were larger when numbers of larvae were used in estimating yield reduction. The percent yield reduction per larva was somewhat greater than the reduction per cavity because more cavities were found in a plant than larvae.

The correlation coefficients between cavities in split stalks and yield and cavities in dissected stalks and yield were similar. This similarity indicates that equally reliable results may be obtained by either the splitting or dissecting method. In comparing Figures 8 and 9, note that the regression lines from data on cavities in split stalks are similar to the lines from data on cavities in dissected stalks. The standard errors of the regression coefficients also indicate little difference between the two methods.

The determination of cavities and larvae by the dissection of stalks is both costly and time consuming and may frequently limit the amount of experimental work that can be properly conducted. For this reason it was hoped that the splitting of stalks, as previously described, would yield results comparable to those obtained from the dissection of stalks. No critical time study was made to show the superiority of splitting stalks versus dissecting stalks. However, it would be safe to state that

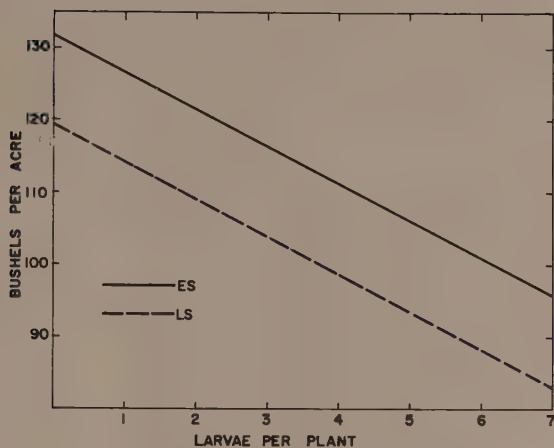


Figure 3. Regression lines for first-brood infestation relating larvae to yield, 1958.

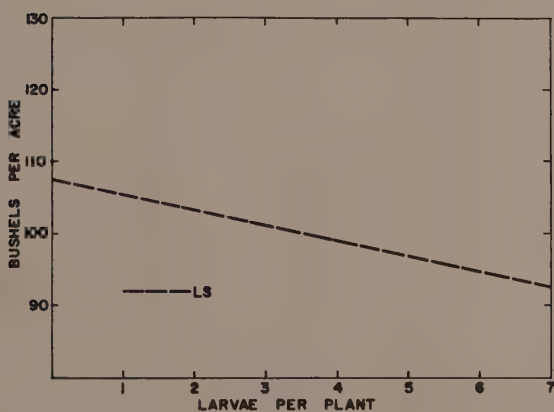


Figure 4. Regression line for second-brood infestation relating larvae to yield, 1959.

Table 4. Data collected in fall from dent corn stalks infested with European corn borer, 1958-59. Average of five replications.

Year	Treatment number	No. of larvae per plant	No. of cavities/plant		Yield Bu./A.
			Split stalks	Dissected stalks	
<u>Early susceptible</u>					
1958	1	0.34	0.72	1.30	128.6
	2	0.72	2.16	2.36	128.4
	3	1.86	4.64	5.46	126.8
	4	0.18	2.42	3.22	123.2
	5	0.68	3.90	4.26	122.3
	6	1.54	5.44	6.24	121.2
	7	0.44	5.14	5.44	110.9
	8	0.78	5.32	5.38	114.0
	9	1.32	6.20	6.86	111.8
1959	1	0	0.12	0.08	110.6
	2	0.20	0.36	0.50	107.9
	3	2.50	6.64	7.42	108.2
	4	0.02	0.50	0.38	112.7
	5	0.16	1.20	1.42	107.7
	6	2.34	6.50	7.40	101.8
	7	0	1.74	2.06	106.6
	8	0.20	2.56	3.34	101.9
	9	2.16	6.80	8.08	100.7
<u>Early resistant</u>					
1958	1	0.18	0.20	0.52	103.5
	2	0.74	1.66	2.20	100.1
	3	1.58	3.76	4.46	101.2
	4	0.38	1.50	1.88	101.7
	5	0.80	2.34	2.60	102.0
	6	1.78	4.74	3.50	99.3
	7	0.38	2.20	2.44	97.9
	8	0.78	3.02	3.34	99.7
	9	1.30	5.26	5.98	98.4
1959	1	0	0.06	0.04	99.8
	2	0.12	0.34	0.42	100.1
	3	1.44	5.00	4.60	98.3
	4	0.02	0.48	0.80	98.4
	5	0.18	0.60	0.98	98.1
	6	1.86	5.06	5.76	99.1
	7	0.02	0.68	1.12	98.8
	8	0.20	0.88	1.14	98.8
	9	1.40	5.16	5.46	96.4

Table 4. (cont.)

Year	Treatment number	No. of larvae per plant	No. of cavities/plant in		Yield Bu./A.
			Split stalks	Dissected stalks	
<u>Late susceptible</u>					
1958	1	0.52	1.72	1.52	120.6
	2	1.38	2.64	3.54	111.8
	3	2.42	5.06	6.28	107.0
	4	0.66	1.34	1.84	117.0
	5	0.88	3.06	3.34	117.6
	6	1.80	5.06	5.64	111.6
	7	0.40	3.72	3.68	104.1
	8	1.08	5.64	5.78	99.7
	9	1.64	6.22	6.82	95.7
1959	1	0.02	0	0.06	107.6
	2	0.26	0.62	0.94	108.1
	3	2.50	6.16	6.58	100.9
	4	0.02	0.04	0.08	109.3
	5	0.50	1.46	1.32	108.4
	6	2.52	5.64	6.88	101.8
	7	0.02	0.42	0.60	103.5
	8	0.22	1.66	1.40	102.8
	9	1.64	5.62	6.12	93.9
<u>Late resistant</u>					
1958	1	0.42	0.58	0.76	99.7
	2	1.30	2.98	3.40	101.9
	3	1.42	3.72	4.36	100.6
	4	0.36	0.72	0.72	103.2
	5	1.22	2.94	3.58	100.4
	6	1.76	3.88	4.70	102.9
	7	0.64	1.82	1.84	100.8
	8	1.08	3.18	3.00	100.0
	9	1.94	4.02	4.92	100.2
1959	1	0	0	0.02	96.3
	2	0.18	1.02	0.54	96.5
	3	1.20	3.70	3.92	92.6
	4	0	0	0.02	97.5
	5	0.30	0.70	0.68	94.1
	6	1.18	3.62	4.66	87.8
	7	0.02	0.12	0.18	95.8
	8	0.30	0.96	0.96	93.3
	9	1.42	4.48	4.76	85.5

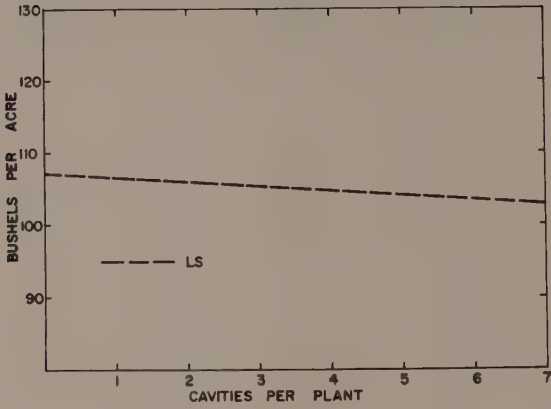


Figure 5. Regression line for second-brood infestation relating cavities in split stalks to yield, 1959.

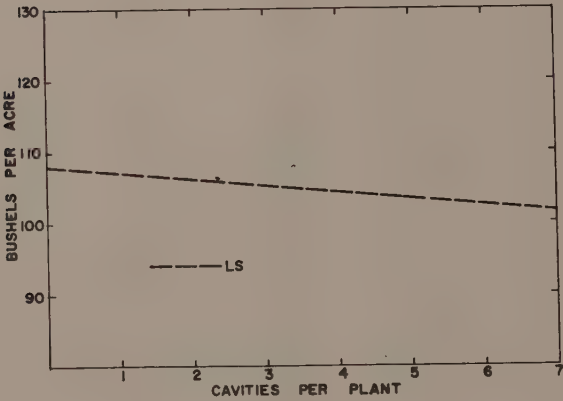


Figure 6. Regression line for second-brood infestation relating cavities in dissected stalks to yield, 1959

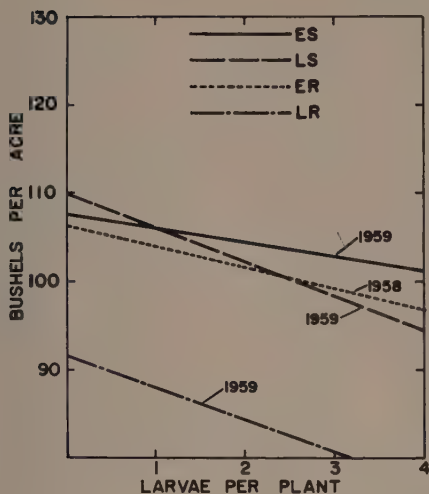


Figure 7. Regression lines for fall dissection relating larvae to yield, 1958-1959.

splitting a stalk requires less than one-fourth the time necessary to dissect a stalk. When data from the 2 years were pooled, the correlation coefficient between the numbers of cavities found in split stalks and those found in dissected stalks was 0.955 with 358 degrees of freedom. This highly significant correlation coefficient indicates that the number of cavities detected by splitting stalks is a reliable estimate of the number of cavities detected by dissecting stalks.

Therefore, the data obtained in this experiment justify the use of cavity counts from split stalks as an index of borer damage. Because splitting a stalk requires less than one-fourth the time necessary to dissect a stalk, it is proposed that this technique be standardized and accepted as a method of determining damage done by the European corn borer. The technique of splitting stalks cannot be used if any data relating to the larval population is desired. Thus information concerning the estimation of future infestation, parasites, or diseases must be obtained by dissecting the stalks and collecting the larvae.

Effect of Two Broods on Yield

The combined effects of the two broods on yield may be demonstrated from the analysis of variance in Table 7. The interaction of the two broods in causing yield reduction was not significant, with the exception of the early susceptible planting in 1958 when the interaction was significant at the 5% level. All other yield differences detected by the analysis of variance were significant at the 1% level. This difference in significance indicates that the effects of second-brood infestation are not greatly modified by a first-brood infestation; thus the total effect of infestation by both broods is a summation of the two without additional effect owing to the combination. Therefore when a first-brood infestation causes reduced yield, the probability of damaging infestations of second-brood borers is greatly decreased.

Table 5. Correlation coefficients of larvae in dissected stalks and of cavities in split and dissected stalks (given separately) with yield. (Data computed from Table 4.)

Year	Hybrid planting	Treatments	Larvae and yield	Cavities in		df
				Split stalks and yield	Dissected stalks and yield	
1958	ES	1, 2, 3	-0.144	-0.234	-0.216	13
		All	0.084	-0.539**	-0.466**	43
	ER	1, 2, 3	-0.464	-0.260	-0.241	13
		All	-0.426**	-0.353*	-0.280	43
	LS	1, 2, 3	-0.371	-0.357	-0.343	13
		All	-0.091	-0.587**	-0.556**	43
	LR	1, 2, 3	0.017	0.006	0.020	13
		All	-0.018	-0.038	-0.027	43
1959	ES	1, 2, 3	0.046	-0.226	-0.182	13
		All	-0.385*	-0.527**	-0.550**	43
	ER	1, 2, 3	-0.333	-0.324	-0.352	13
		All	-0.152	-0.230	-0.252	43
	LS	1, 2, 3	-0.658**	-0.685**	-0.705**	13
		All	-0.583**	-0.715**	-0.698**	43
	LR	1, 2, 3	-0.335	-0.288	-0.394	13
		All	-0.491**	-0.554**	-0.585**	43

*Significant at the 5% level

**Significant at the 1% level

When the results given in Tables 3 and 6 are compared, it is readily seen that the most reliable estimates of yield loss were obtained when the first-brood dissection data were used. First-brood infestation resulted in considerably more yield loss than did second-brood infestation. At the time of the fall dissection it was impossible to distinguish whether a given cavity was made by a first- or a second-brood borer. If the amount of yield loss is based on cavities found in the fall, the reduction in yield will be overestimated if the majority of the cavities were the result of second-brood infestation, and underestimated if the majority of the cavities were the result of first-brood infestation. In computing results from fall dissections the use of larvae would have given even less reliable results than cavity counts because the majority of those larvae belonged to the second brood. Very few first-brood larvae, which actually cause the greatest loss in yield were found in the fall. Thus the standard estimate of "3% loss per borer per plant," based on the number of larvae found in the fall, is not an accurate measure of borer damage.

Table 6. Regression equations from fall dissections relating yield of dent corn in bushels per acre and yield loss to larvae, cavities in split stalks, or cavities in dissected stalks. (Data computed from Table 4.)

Year	Hybrid planting	Treatments	Regression equation	Standard error of regression coefficient	df	Yield loss/unit of damage (%)
<u>Larvae and yield</u>						
1958	ER	All	$Y = 106.2 - 2.09X$	0.69	43	2.0
1959	ES	All	$Y = 107.9 - 1.64X$	0.60	43	1.5
	LS	1, 2, 3	$Y = 107.5 - 2.17X$	0.44	13	2.0
	LS	All	$Y = 106.6 - 3.04X$	0.65	43	2.8
	LR	All	$Y = 95.7 - 4.74X$	1.28	43	4.9
<u>Cavities in split stalks and yield</u>						
1958	ES	All	$Y = 129.9 - 2.28X$	0.54	43	1.7
1959	ER	All	$Y = 102.3 - 0.67X$	0.26	43	0.6
	LS	All	$Y = 120.8 - 3.00X$	0.57	43	2.5
	ES	All	$Y = 109.2 - 0.91X$	0.22	43	0.8
	LS	1, 2, 3	$Y = 107.5 - 0.86X$	0.25	13	0.8
	LS	All	$Y = 107.9 - 1.60X$	0.24	43	1.5
	LR	All	$Y = 96.3 - 1.87X$	0.42	43	1.9
<u>Cavities in dissected stalks and yield</u>						
1958	ES	All	$Y = 129.3 - 1.91X$	0.55	43	1.5
1959	LS	All	$Y = 120.0 - 2.52X$	0.57	43	2.1
	ES	All	$Y = 109.3 - 0.82X$	0.19	43	0.7
	LS	1, 2, 3	$Y = 107.8 - 0.90X$	0.25	13	0.8
	LS	All	$Y = 111.2 - 2.68X$	0.21	43	2.4
	LR	All	$Y = 96.3 - 1.75X$	0.38	43	1.8

Table 7. Analysis of variance of yield data pertaining to European corn borer infestation of dent corn, 1958-59.

Source of variation	df	Mean squares		F-value	
		1958	1959	1958	1959
<u>Early susceptible</u>					
Replications	4	114.852	12.192		
First brood effect	2	946.450	127.080	84.550**	11.042**
Second brood effect	2	10.689	148.286	<1	12.884**
First brood:second brood interaction	4	42.782	29.921	3.822*	2.610
Experimental error	32	11.194	11.509		
Total	44				
<u>Early resistant</u>					
Replications	4	35.292	12.786		
First brood effect	2	36.374	7.532	2.489	<1
Second brood effect	2	7.174	5.512	<1	<1
First brood:second brood interaction	4	11.184	4.848	<1	<1
Experimental error	32	14.612	9.616		
Total	44				
<u>Late susceptible</u>					
Replications	4	9.354	9.150		
First brood effect	2	1043.723	181.011	37.529**	20.931**
Second brood effect	2	298.943	372.313	10.749**	43.052**
First brood:second brood interaction	4	21.143	14.999	<1	1.734
Experimental error	32	27.811	8.648		
Total	44				
<u>Late resistant</u>					
Replications	4	12.150	68.244		
First brood effect	2	12.973	50.237	3.179	2.682
Second brood effect	2	0.904	256.168	<1	13.677**
First brood:second brood interaction	4	8.815	12.319	2.160	<1
Experimental error	32	4.080	18.730		
Total	44				

*Significant at the 5% level

**Significant at the 1% level

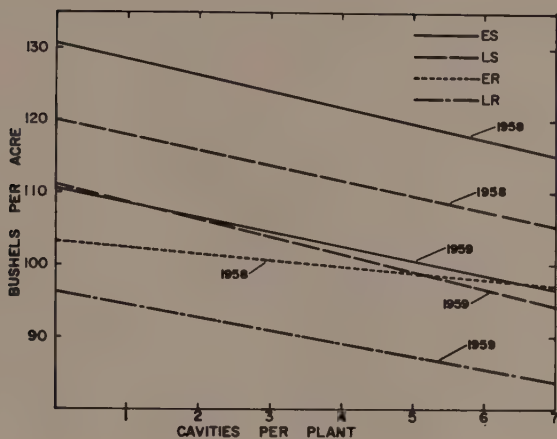


Figure 8. Regression lines for fall dissection relating cavities in split stalks to yield, 1958-59.

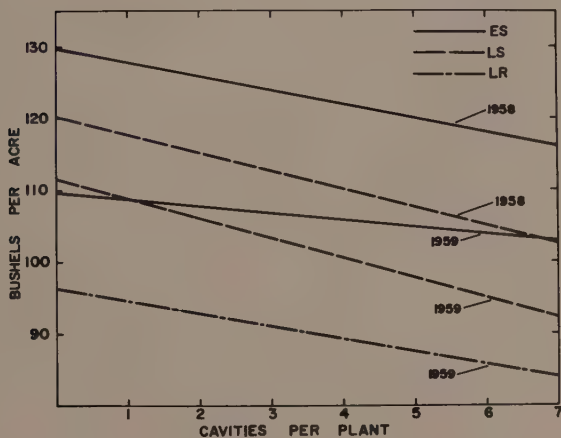


Figure 9. Regression lines for fall dissection relating cavities in dissected stalks to yield, 1958-59.

SUMMARY

Data were collected at Ankeny, Iowa, in 1958 and 1959 to determine the effect of the European corn borer on the yield of dent corn.

First-brood infestation resulted in a greater yield loss than did infestation by the second brood. Both cavities and leaf lesions were a better index of damage than were larvae. A greater yield reduction per unit of damage occurred in the late planting than in the early planting, indicating that smaller plants were more severely damaged by a first-brood infestation than were the larger plants.

In determining second-brood infestation, and infestation by both broods, cavities in both split and dissected stalks gave a better estimate of borer damage than did larvae. Because results from split or dissected stalks are equally reliable, and because splitting requires about one-fourth the time required to dissect a stalk, it is proposed that stalk splitting be adopted as a standard procedure in determining borer damage.

When yield loss occurs, the reduction is due to either infestation by first- or second-brood larvae, or a summation of the two, without an additional effect due to the interaction of infestation by both broods.

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COMPARISONS OF THE IOWA FOREST RESOURCE IN
1832 and 1954

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ABSTRACT. The forest area in Iowa by counties obtained from the Original United States Land Office Survey, 1832-1859, is compared with Forest Survey data of 1954. This paper serves to make as matter of record the data obtained from the earlier survey by planimetric measurement. The approximate decrease of forested land has been 61%.

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Iowa is known as one of the richest agricultural states in the Nation. The rise to agricultural prominence was preceded by substantial changes in the forests of Iowa including timber depletion and land clearing. Originally, approximately 18% of the land area in the state was forested. Today, about 7% is forested.

Historical accounts of the early forest resource in Iowa are meager. Original estimates identified forests as occupying from less than one-eighth (3) to one-fifth (2) of the total land area of the state. The most reliable estimates appear to be taken from planimetric measurement of maps based on the Original United States Land Office Survey, 1832-1859, recorded in typed memorandum at the Iowa State University Forestry Department, Ames (1). These data are shown by county in Table 1 with similar data from the Forest Survey of Iowa, 1954 (4).

It would be improper to consider the forest cover changes exactly as depicted here because the definitions of what is and is not forest land has changed. In the Original Survey the surveyors were actually on section lines only, that is, they girded the state with squares one mile on a side. In the 1954 Survey forest land was interpreted from aerial photographs supported by field observation and the averages were a result of defining the forest only at certain points. The latter Survey was a sample designed to give reasonably accurate acreages for rather large segments of the state, and each county was not measured with equal precision. These figures, however, do portray an interesting change in the appearance of the state.

There are some interesting historical aspects of the Original Land Survey in Iowa. This was part of the first surveys in Michigan, Wisconsin and Iowa Territories when all basic points, sectional lines and township lines were established. Edgar Harlan, state historian, reports the

Table 1. Forest area in Iowa by county, 1832-1859 and 1954.

County	Forest Area (acres)		Per cent Decrease
	1832-1859	1954 ¹	
1. Adair	32,768	12,000	63
2. Adams	36,147	16,000	44
3. Allamakee	376,220	132,000	65
4. Appanoose	133,760	56,000	58
5. Audubon	13,516	4,000	70
6. Benton	64,204	20,000	69
7. Black Hawk	49,280	17,000	25
8. Boone	62,080	30,000	52
9. Bremer	47,360	15,000	68
10. Buchanan	64,307	17,000	74
11. Buena Vista	1,200	5,000	317 ²
12. Butler	39,680	15,000	62
13. Calhoun	3,000	2,000	33
14. Carroll	10,320	5,000	52
15. Cass	30,720	9,000	71
16. Cedar	76,000	23,000	70
17. Cerro Gordo	21,760	4,000	82
18. Cherokee	5,720	11,000	92 ²
19. Chickasaw	85,500	16,000	81
20. Clarke	55,560	39,000	30
21. Clay	3,300	8,000	142 ²
22. Clayton	366,340	120,000	67
23. Clinton	80,896	30,000	63
24. Crawford	10,810	14,000	30 ²
25. Dallas	64,640	36,000	44
26. Davis	200,640	51,000	75
27. Decatur	126,000	57,000	55
28. Delaware	111,615	27,000	76
29. Des Moines	125,340	40,000	68
30. Dickinson	1,980	4,000	102 ²
31. Dubuque	201,825	56,000	72
32. Emmet	4,000	4,000	00
33. Fayette	126,770	38,000	70
34. Floyd	62,800	9,000	86
35. Franklin	16,000	4,000	75
36. Fremont	57,139	31,000	46
37. Greene	25,440	12,000	53
38. Grundy	640	1,000	56 ²
39. Guthrie	44,032	38,000	14
40. Hamilton	19,520	9,000	54
41. Hancock	8,960	3,000	67
42. Hardin	43,520	14,000	68
43. Harrison	64,380	44,000	32
44. Henry	114,995	36,000	69
45. Howard	51,920	11,000	79
46. Humbolt	4,800	6,000	25 ²
47. Ida	640	2,000	212 ²
48. Iowa	90,315	30,000	67

Table 1. (cont.)

County	Forest Area (acres)		Per cent Decrease
	1832-1859	1954 ¹	
49. Jackson	282,420	82,000	71
50. Jasper	68,800	31,000	55
51. Jefferson	143,250	37,000	74
52. Johnson	108,545	41,000	62
53. Jones	136,705	42,000	69
54. Keokuk	116,531	35,000	70
55. Kossuth	3,840	8,000	108 ²
56. Lee	179,100	81,000	55
57. Linn	153,600	46,000	70
58. Louisa	101,065	41,000	59
59. Lucas	64,640	51,000	21
60. Lyon	1,000	4,000	300 ²
61. Madison	72,800	50,000	18
62. Mahaska	111,360	31,000	72
63. Marion	131,060	52,000	60
64. Marshall	32,320	14,000	57
65. Mills	50,790	25,000	51
66. Mitchell	66,355	10,000	85
67. Monona	49,130	48,000	2
68. Monroe	86,400	71,000	18
69. Montgomery	36,864	10,000	73
70. Muscatine	90,820	30,000	70
71. O'Brien	1,500	4,000	167 ²
72. Osceola	640	2,000	212 ²
73. Page	51,200	12,000	77
74. Palo Alto	5,600	6,000	71 ²
75. Plymouth	3,640	12,000	230 ²
76. Pocahontas	2,200	1,000	55
77. Polk	67,200	32,000	52
78. Pottawattamie	51,814	27,000	48
79. Poweshiek	33,600	17,000	49
80. Ringgold	50,030	27,000	46
81. Sac	2,200	6,000	173 ²
82. Scott	43,000	15,000	65
83. Shelby	16,690	5,000	70
84. Sioux	700	3,000	329 ²
85. Story	37,440	13,000	65
86. Tama	79,680	30,000	62
87. Taylor	57,036	21,000	63
88. Union	28,800	22,000	24
89. Van Buren	201,730	64,000	68
90. Wapello	145,280	49,000	66
91. Warren	82,640	44,000	47
92. Washington	94,412	37,000	61
93. Wayne	56,440	27,000	52
94. Webster	46,080	26,000	44
95. Winnebago	5,120	3,000	41
96. Winneshiek	152,780	56,000	63

Table 1. (cont.)

County	Forest Area (acres)		Per cent Decrease
	1832-1859	1954 ¹	
97. Woodbury	19,860	25,000	26 ²
98. Worth	9,220	5,000	46
99. Wright	<u>8,640</u>	<u>6,000</u>	<u>31</u>
Totals	6,680,926	2,620,000	Av. 61

¹ Reported in thousands of acres

² Per cent increase in forest land

Survey was begun March 23, 1932, and completed 27 years later on August 17, 1859. It cost 13 million dollars.

The first survey point in Iowa was established by Jennifer T. Sprague at 10:00 a.m., March 23, 1832, after he arrived March 15, 1832, by barge from Ohio. Mr. Sprague's original notes, which are quite complete and very original, are in vault at the Securities Department, State House, Des Moines, Iowa.

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THE ROLE OF NONBONDED REPULSIONS IN SECONDARY
ISOTOPE EFFECTS. II. INFLUENCE ON STRUCTURE;
GAMMA AND MORE DISTANT EFFECTS¹

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ABSTRACT. The model presented in paper I to describe secondary α and β deuterium effects is extended to the treatment of nonbonded contributions in a variety of other secondary isotope effects. Gamma and more distant effects are discussed, including effects of solvation. In the small p-methyl effect found by Lewis the model fails unless charge is delocalized from the methyl hydrogens. Nevertheless, according to the model, if charge is delocalized the relief of nonbonded repulsions is as important in the isotope effect as the hyperconjugative weakening of C-H bonds. It is shown how the model, in its harmonic, nondipolar approximation, leads to some of the same conclusions as Halevi's anharmonic, inductive model. Predictions are made concerning isotope effects involving γ deuteriums, rotational isomerization energies, steric inhibition of resonance, and lengths of bonds adjacent to deuterium.

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INTRODUCTION

In paper I of this series (1) a method was outlined for calculating contributions of nonbonded repulsions in secondary α and β deuterium isotope effects. The qualitative success of the approach justifies the testing of its application to other types of secondary isotope effects. It is appropriate before further application to consider briefly the relation of the present model to conventional analyses of secondary isotope effects. For simplicity in the discussion we shall neglect anharmonicity, as is customarily done, although it will be referred to later in the paper.

Molecular force fields, to a rather good approximation, can be represented by the Urey-Bradley field which is the sum of "valence" (stretching and bending) and "nonbonded" interactions. Molecular vibrational

¹ Research supported by the National Science Foundation. Presented at the 138th Meeting of the American Chemical Society, New York, New York, September 12, 1960.

frequencies and their shift with isotopic substitution are uniquely determined by the molecular force field, and if all the relevant frequencies are known it is possible to compute zero-point energy differences and secondary isotope effects on rates and equilibrium constants (2). Almost all practical treatments so far have used frequencies determined empirically for model compounds or observed rate data, interpreted results in terms of valence force fields, and then invoked specialized interactions (as hyperconjugation, hybridization, induction, etc.) to explain shifts in valence force constants. The present work starts from a different point of view. It examines the extent to which the nonbonded components of the field, which change dramatically as the degree of crowding in molecules is altered in reactions, can account for observed isotope effects. To do this a scheme has been devised to circumvent the formidable problem of computing frequencies from complex force fields directly and it is shown how to estimate hydrogen atom nonbonded contributions to zero-point energies even if a detailed knowledge of the force field is unavailable. The most important consequence of the model is that it is found to account qualitatively or semiquantitatively for many effects which have almost universally been attributed to the more specialized interactions referred to above.

According to the model a reactant (I) is transformed into a transition state or product (II) which, in the normal isotope effect, is sterically relieved with respect to the reactant. In inverse isotope effects the hydrogens of (II) may be more crowded than those of (I). An expression was derived in paper I for the nonbonded contribution to the difference, $\Delta\Delta E$, in activation energy or reaction energy. For the difference between deuterated and protonated species $\Delta\Delta E$ corresponds to a sum over all pairs of atom-atom interactions, $\Delta\Delta E_{ij}$, in which deuterium is substituted for hydrogen. Each interaction, $\Delta\Delta E_{ij}$, reduces, in the harmonic approximation, to

$$\Delta\Delta E_{ij} \simeq 0.135(l_m^2)_{ij} [1 + 15 \times 10^{16}(l_t^2)_{ij}] [V''_{ij}(r_g \text{ II}) - V''_{ij}(r_g \text{ I})], \quad (1)$$

very nearly, where l_t is expressed in cm. The quantities l_m^2 and l_t^2 , respectively, are mean-square hydrogen and mean-square hydrogen-plus-skeletal amplitudes of vibration of the ij^{th} pair, for which the magnitudes are reasonably well known and listed in paper I. The quantities V''_{ij} are second derivatives of the nonbonded potentials evaluated at the appropriate mean internuclear distances, r_g , of (I) and (II). The values of the V''_{ij} are only roughly known but tentative plots against r_g are given in paper I. Since the V''_{ij} are positive for small r_g and rapidly decrease as r_g increases, it is easily seen from Eq. (1) that $\Delta\Delta E_{ij}$ is smaller for hydrogen than deuterium in reactions where the r_g increase (i.e. $\Delta\Delta E_{ij}$ is negative). Thus, hydrogens, with greater amplitudes of vibration than deuteriums, behave as if they were bulkier and drive the reaction more vigorously than the more massive deuteriums. Tritium-hydrogen $\Delta\Delta E$ values would be expected to be about 40% greater than deuterium-hydrogen values, according to the influence of inertia on zero-point amplitudes of vibration.

Further considerations will be introduced in the following to account for effects of charge, anharmonicity, and rate of change of nonbonded

energy with distance. At the present state of development it is impractical to carry out calculations that are both rigorous and all-embracing, and many of the following calculations are conspicuously oversimplified. Nevertheless it is worthwhile to get an idea of the over-all scope of the model at the outset of the investigation.

SUBSTITUTION MORE DISTANT THAN BETA CARBONS

Deuteriums and Reaction Site Separated by Benzene Ring

The effect of deuterium and tritium substitution on methyl groups attached to aromatic rings has been investigated experimentally by several workers (3, 4) with a view to examining hyperconjugative isotope effects analogous to those presumed to occur in the β effect. As discussed in paper I the present model accounts in molecular ground states and in transition states in β effects for many results usually attributed to hyperconjugation. It is interesting that the present model in its approximation neglecting charge leads to a different prediction for aromatic substitution than the hyperconjugation model. It suggests an essentially null isotope effect in the aromatic substitution of toluene since the steric environment of the methyl group is almost unchanged in the reaction. This is compatible with the smallness of the effect found by Swain and Kresge (3) in nitration, mercuriation, and bromination of toluene where resonance effects such as hyperconjugation would be expected to be significant. Likewise the model would imply a null effect in the solvolysis of p-methyltolylcarbonyl chloride. In this case, however, Lewis has reported a small effect (~ 16 cal/D at most) depending very strongly on the solvent (4). Highly nucleophilic solvents were found to wipe it out entirely. While our model fails here in its uncharged approximation, it can be applied to make rough estimates not only of deuterium retardation but also of the effect of the solvent if the charge is taken into account.

Let us first consider the effect of the reduction of the nearest nonbonded $C \cdots H$ repulsions attending the partial loss of a $2p\pi$ electron from the para carbon of the ring, as estimated from Mulliken's magic formula (5) (renormalized slightly to the present potential functions). This, in principle, would be subject to an isotope effect entirely analogous to the foregoing effects. If it is assumed that the time-average deficit of charge at the carbon is 0.2_3 electrons (6), the calculated result for the isotopic vibrational amplitude effect is only $\Delta\Delta E_a \sim 2$ cal/D. The smallness of this effect is a result of the approximate orthogonality of the hydrogen $1s$ and carbon $2p\pi$ orbitals, which overlap appreciably in charge distribution. While it is possible that the effect of orthogonality on nonbonded interactions is overestimated by the magic formula, the above calculation is not convincing support that electronic depletion at the p-carbon atom is responsible for the isotope effect. Partial delocalization of the charge onto the hydrogens, on the other hand, gives a calculated nonbonded isotope effect an order of magnitude greater than that of the above result, owing to diminution of both $H \cdots H$ and $H \cdots C$ interactions. In this case orbital orientations do not detract appreciably from the isotope effect.

If we assume that reduction of nonbonded repulsions and of covalent C-H bond order, as implied by resonance forms with ionized p-methyl

hydrogens, is in proportion to the time-average loss of charge by the hydrogens,² it is possible to estimate $\Delta\Delta E_a$. The loss of one electron would cause one V_{CH}'' and two V_{HH}'' to vanish; so for the loss of \underline{n} electrons we obtain, according to Eq. (1),

$$\Delta\Delta E_a = 0.135n \left\{ 2[(1+15 \times 10^{16} l_t^2) l_m^2 V'']_{HH} + [(1+15 \times 10^{16} l_t^2) l_m^2 V'']_{CH} \right\}.$$

The result for the V'' determined for normal bond distances and angles gives $\Delta\Delta E_a$ (nonbonded) $\sim 550 n$ cal per 3D or
 $\sim 180 n$ cal/D

Further, we assume the C-H bond is represented by a Morse-like potential with an asymmetry constant, \underline{a} , as usual, more or less independent of the dissociation energy, D . Then the force constant, k , is proportional to D , and is diminished by the loss of \underline{n} electrons from all three bonds (i.e. $n/3$ per bond) by the ratio $(1-n/3):1$. Since the zero-point energy is proportional to $k^{1/2}$, for small \underline{n} we obtain the result

$$\begin{aligned} \Delta\Delta E_a \text{ (stretch)} &\sim (n/6) [h(\nu_H - \nu_D)/2] \\ &\sim 190 n \text{ cal/D,} \end{aligned}$$

where the ν_i pertain to the stretching frequencies in the reactant. Accordingly, we see that the effect of the relief of nonbonded repulsions due to charge delocalization in the carbonium ion is of the same magnitude as that of the "bond weakening," and that a deficiency of less than 1/20 of an electron distributed over the hydrogens in the p-methyl group can account for the observed isotope effect. A coupling of the results of calculations of Brickstock and Pople (6) with those of Muller and Mulliken (7) suggests a similar magnitude for the shift of charge. The β isotope effect may also be subject to this shift, as suggested in I.

Since delocalization of charge from methyl groups is the sine qua non of hyperconjugation theory, the foregoing treatment of the p-methyl effect amounts, more or less, to invoking hyperconjugation. The implication that nonbonded relief may be as important as bond weakening is, however, new.

In treating the solvent effect, let us consider the ion-dipole interaction at the site of the para carbon in the ring. Since the lateral amplitude of vibration of p-methyl hydrogens is about 0.014 Å greater than that of deuteriums, a solvent molecule, crowded toward the methyl by solvent molecules adhering to other positive sites, might be held further away from the carbonium ion by an amount up to 0.014 Å.³ For sake of argument let us take the solvent dipole to be the carbonyl group in acetone

² This simple relation between charge and covalent bonds is more or less compatible with the calculations of N. Muller and R.S. Mulliken (7).

³ A calculation based on van der Waals forces and harmonic vibrations suggests that the isotopic difference, δr , in molecular separations is actually more nearly $[l_m^2(H) - l_m^2(D)] V''^{1/2} V'' \sim [l_m(H) - l_m(D)] / 2$ than $[l_m(H) - l_m(D)]$. In the case above, however, the asymmetry of vibrations would enhance δr by roughly $[l_m(H) - l_m(D)] / 3$.

with charges localized on the C and O atoms, separated by the bond distance, r_2 . Further, let the distance between the nuclei of the ring carbon and the solvent oxygen atom be r_1 , the charge at the para carbon be $n\epsilon$, where ϵ is the electronic charge, and the carbonyl bond moment be $m\epsilon r_2$. The decreased stability per solvent molecule attached, δV , is related to the increased separation, δr , of r_1 owing to hydrogen vibrations, assuming coulombic interactions, by

$$\delta V \simeq mn\epsilon^2 [r_1^{-2} - (r_1 + r_2)^{-2}] \delta r.$$

If we assign the values $m = 0.4_5$ and $n = 0.2_3$ electrons (6), $r_1 = 3.0\text{\AA}$, $r_2 = 1.2\text{\AA}$, and $\delta r = 0.014\text{\AA}$, then the result for δV is about 26 cal/mole, the approximate magnitude of the solvent effect. Presumably the effect would be smaller the less nucleophilic the solvent, in agreement with Lewis' empirical observations (3). The model could also account for the slight rate enhancement by *m*-methyl deuteriums. It is possible that appreciable variations in mechanism from solvent to solvent are much more important than the simple ion-dipole effects.

Gamma Substitution Effects

If α and β deuterium substitution effects are steric in origin, it should be possible in very crowded molecules to observe γ effects. For example, if methyl groups were replaced by *t*-butyl groups in the alkyl chlorides considered in paper I, some of the resulting $\text{H}\cdots\text{Cl}$ distances would be several tenths of an angstrom unit shorter than the original methyl $\text{H}\cdots\text{Cl}$ distances if bond angles were considered to remain tetrahedral and bond distances were normal. Such a shortening would contribute the order of 50 cal/mole of interactions to $\Delta\Delta\text{EHCl}$ if a classical carbonium ion were formed. It is possible that distortion exactly analogous to that encountered in $(\text{CH}_3)_3\text{B}:\text{N}(\text{CH}_3)_3$ in paper I would lower the isotope effect appreciably, as it does in the boron compound. Even if the molecule were appreciably deformed the effect would appear to be measurable and (if it existed) not convincingly related to hyperconjugation. If more than one *t*-butyl group were joined to the α carbon a steric γ isotope effect would be enhanced.⁴

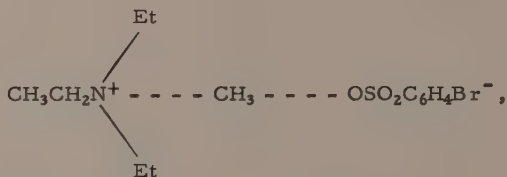
If the transition states in the compounds presently considered turn out not to be well represented by free carbonium ions with conventional structures, the predicted γ isotope effects will necessarily be in error. Such a situation appears to occur in a small γ isotope effect opposite in direction to the one suggested above, which has been observed in the solvolysis of several *n*-propyl derivatives by Leffek, Llewellyn and Robertson (9).

⁴ Since the original proposal of the present nonbonded model (8) there has been increased activity in the study of secondary isotope effects with substitution more remote than the β carbon. It is well to observe that if the number of carbons separating the hydrogen and the leaving group, X , is greater than two or three, the purely steric effect may be very small even if standard molecular models suggest an unusually close $\text{H}\cdots X$ distance. This is because the skeletal flexibility increases substantially with increasing links in the chain unless unusual constraints are imposed, and the steric effect vanishes rapidly with modest increases of flexibility.

The transition state proposed by Robertson et al is quite different from that assumed in the present calculations involving the more crowded tertiary derivatives. Steric hindrance of the end methyl by the leaving group was invoked, a scheme formally in harmony with the present steric arguments for the particular geometry assumed by Robertson et al.

HALEVI'S INDUCTIVE EFFECT

Halevi (10,11) has observed secondary deuterium isotope effects in ionization constants of acids and bases which he has interpreted in terms of a greater electron release by D than by H. Using this difference in inductive power as a basis, Halevi (11) and Lewis (4) have advanced arguments to account for effects formally opposite to hyperconjugative effects. For example, in the solvolysis of methyl parabromobenzene-sulfonate by triethylamine, Lewis observed that deuterium substitution in the amine led to an appreciable enhancement of the rate (4). It may be noted that while the isotope effect is opposite to that based on the hyperconjugative argument if the transition state is considered to be



the effect is in just the direction expected according to the present non-bonded repulsion model. According to the nonbonded model, an analogous case would be the amine-borane equilibrium discussed in paper I, if the amine were deuterated instead of the borane.

Studies have shown that deuterium substitution can lead to measurable differences in dipole moments (12,13). In simple molecules such as HCl these are apparently closely related to the appreciable differences between hydrogen and deuterium mean positions resulting from the above mentioned differences between amplitudes of vibration in the anharmonic potential well. In more complex molecules, as will be explained later, not only may the hydrogen distances be shifted by deuterium substitution, but also the distances and angles of other nearby bonds. Such distortions, which would occur even for harmonic hydrogen vibrations, no doubt have some influence on dipole moments and even nuclear magnetic resonances (14), where chemical shifts have been quoted as supporting the inductive model (14,15). There seems to be no doubt that actual isotopic differences in bond polarities may indeed result from anharmonicity of vibration. We should like to suggest, however, that not all effects contrary to the hyperconjugation model need be attributed solely to the anharmonic dipole effect. The present model, even in its nondipolar, harmonic approximation, can account for many of the same effects.

ADDITIONAL IMPLICATIONS OF MODEL

Rotational Isomerization

Closely akin to a γ effect would be an isotopic alteration of the difference in energy, ΔE_{tg} , between trans and gauche conformations of *n*-butane. According to the present model, ΔE_{tg} would be about 60 cal/mole or 50 cal/mole less, respectively, for $D_3C-CH_2-CH_2-CD_3$ or $D_3C-CD_2-CD_2-CD_3$ than for $H_3C-CH_2-CH_2-CH_3$ if the structural parameters of the various isotopic species were assumed to be identical. Since the most accurate existing value for the normal species is 770 ± 90 cal/mole (16) (for the liquid), the detection of such a small effect might not be simple. An experimental investigation would seem worthwhile, nevertheless, in view of its possible utility in the interpretation of isotope effects. It is not difficult to think of other isomerizations which should show analogous effects.

Steric Inhibition of Resonance

There are many well-known physical and chemical manifestations of steric effects (17,18), which, according to the present model, should presumably be subject to isotope effects since H atoms may be regarded as having a slightly larger size than D atoms. Unless the steric manifestations were large and sensitive to energy changes of tens of calories, however, the isotope effects would be obscured. It is useful to consider possible magnitudes to be expected in an extensively investigated case, the steric inhibition of resonance.

It has often been observed in substituted benzenes, that when groups with π electrons interacting with the benzene ring are twisted out of planarity with the ring by steric forces, significant alterations of dipole moments, electronic spectra, and acid strengths, for example, may occur. Since the isotopic steric influence on the twist angle is likely to be only the order of 0.3° in the most favorable cases, it is unlikely that corresponding shifts in relatively insensitive quantities as dipole moments would be significant.

Effects on electronic spectra might be barely measurable. The variation with twist of molar extinction coefficients in the ultraviolet spectra of *o*-substituted *N,N*-dimethylanilines has been studied by Kleven and Platt (19). In the most striking bands the variation was nearly an order of magnitude as the twist went from 0 to 90° . Hence, deuterating ortho-methyls and/or the *N*-methyls might lead to an enhancement of extinction coefficients of up to a few tenths of a percent. Similarly, if the ortho hydrogens in biphenyl were replaced by deuterium, the rings could approach coplanarity more closely by perhaps 0.5° ⁵ and increase

⁵ It is possible, using the potentials of reference 1 and an angularly dependent π electron potential of the form $V_\pi^0 \cos^2\theta$, to calculate the approximate equilibrium angle of twist between the planes of the benzene rings and the isotopic shift in twist angle. For sake of illustration V_π^0 was taken to be 5 kcal/mole. It is interesting to note that the calculated twist of roughly 38° is in reasonable agreement with the experimental electron diffraction result of $42^\circ \pm 10^\circ$ [O. Bastiansen, Acta Chem. Scand., 2:408 (1949)]. A 50% change in V_π^0 changes the calculated twist by only about 5° .

the extinction coefficient slightly. Vibrational perturbations of the electric wave functions, however, are also mass-sensitive and might easily obscure the steric effect (20).

One of the clearest influences of steric inhibition of resonance on acid strengths has been found by Wheland, Brownell, and Mayo (21) in the case of substituted p-nitrophenols. Substitution of methyls at both positions ortho to the nitro group reduces the acid dissociation constant ten-fold. We might expect deuteriation of the methyl groups to reduce steric strain and augment the dissociation constant. The smallness of the change in size on deuterating, however, again suggests an isotope effect of only a few tenths of a percent. This seems no larger than the anticipated solvent effect, analogous to that discussed earlier, which should act in the same direction. It may also prove to be smaller than Halevi's inductive effect which should act in the opposite direction. We must tentatively conclude that isotope effects associated with steric inhibition of resonance are likely to be very small and masked by other factors.

Molecular Structure

Another effect implied by the present model is an isotopic alteration of lengths of bonds between atoms, one of which is adjacent to hydrogen or deuterium. For example, the C-C bond length in C_2H_6 should be larger than in C_2D_6 , owing to the greater vibrational enhancement of the force between C...H's across the C-C single bond than between C...D's. The treatment of averaging interatomic forces, f_{ij} , over molecular vibrations is exactly analogous to that of averaging potentials and, neglecting anharmonicity, we may use Eq.(1) if V_{ij}'' is replaced by its derivative, getting

$$\begin{aligned}\Delta f_{ij}(\text{harm}) &= [\bar{f}_H(r_g) - \bar{f}_D(r_g)]_{ij} \\ &\simeq -0.13_5 l^2(H) [1 + 15 \times 10^{16} l^2(H)] V_{ij}'''(r_g).\end{aligned}\quad (1)$$

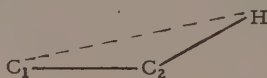
Because of the geometry of the interactions in ethane, however, it is apparent that stretching anharmonicity effects will tend to be much larger than in the cases previously considered, and will act to diminish the isotope effect. The anharmonic contribution to the isotope force difference, $\Delta f_{ij}(\text{anh})$, arising because the mean distance, r_g , is greater for C...H than C...D, can be computed by the relation

$$\begin{aligned}\Delta f_{ij}(\text{anh}) &\simeq \left\{ \left(\frac{\partial f}{\partial r} \right) [r_g(H) - r_g(D)] \right\}_{ij} \\ &= -[V''(r_g) \Delta r]_{ij}\end{aligned}\quad (2)$$

where $\Delta r = r_g(H) - r_g(D)$. The term Δr is related to Δr_{CC} , the result sought, and Δr_{CH} , the difference between the C-H and C-D mean bond lengths, according to

$$\Delta r \simeq \Delta r_{CC} \cos \beta + \Delta r_{CH} \cos \gamma \quad (3)$$

where, in the triangle



$\beta = \angle C_2-C_1 \cdots H$ and $\gamma = \angle C_2-H \cdots C_1$. The difference, Δr_{CH} , is known for methane to be 0.005\AA (22) and it is probable that the result for ethane is nearly the same. The final relation needed for the estimation of Δr_{CC} in ethane, coming from the definition of the force constant, is

$$\Delta r_{CC} = \Delta f_{\text{net}}/K_{CC} \quad (4)$$

where

$$\Delta f_{\text{net}} = \sum [\Delta f_{ij}(\text{harm}) + \Delta f_{ij}(\text{anh})] \cos \beta \quad (5)$$

and K_{CC} is the force constant for the C-C bond. For simplicity the C-C-H angles have been assumed to be the same as the C-C-D angles. In principle it should be possible to calculate an isotopic shift of angles, also.

If bond distances, angles, and amplitudes are assumed to be the same as in the alkanes n-butane through n-heptane (23), and the potentials implicit in the curves of Fig. 1 of I are used, it is found that $\Delta f(\text{anh})$ is nearly 40% of $\Delta f(\text{harm})$, and that $\Delta r_{CC} \sim 0.003\text{\AA}$. This shift, while not large, is large enough to be measured by modern electron diffraction methods. If it were confirmed it would have important consequences in spectroscopic analyses of molecular structure where conventional methods of isotopic substitution would be found to compound the discrepancy.

CONCLUSIONS

Secondary isotope effects have been studied intensively in recent years, with special emphasis on the inference of the nature of the transition state in reactions. We have shown that nonbonded interactions may give an appreciable contribution to secondary isotope effects in a variety of reactions, including those which have been previously interpreted on the basis of more specialized interactions such as hyperconjugation. It has been possible to relate the changes in zero-point energies of groups with changes in the degree of crowding in molecules by a simple equation. The direct numerical relationship proposed between nonbonded contributions to isotope effects and the closeness of approach of nonbonded atoms may prove useful in assessing steric factors in reactions, perhaps even in enzymatic reactions which involve considerable steric specificity.

The present model also led to the prediction of several isotope effects that have not yet been studied.

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EXCHANGE OF PHOSPHORUS BETWEEN ORTHOPHOSPHATE
AND CONDENSED PHOSPHATES IN VITRO AND IN SOIL¹R.R. Allmaras, Norio Koiwa, and C.A. Black²

SUMMARY. Evidence was found for slight exchange of P between orthophosphate and the condensed phosphates from fertilizer-grade calcium metaphosphate. The evidence was obtained by tagging the orthophosphate with P^{32} , allowing the orthophosphate and condensed phosphates to interact at room temperature, separating the orthophosphate from the condensed phosphates by paper chromatography, and measuring the specific activity of the condensed phosphate. Where the orthophosphate and condensed phosphates were present in solution with no additives, less than 1 per cent of the radioactivity was associated with the condensed phosphates after a 72-hour equilibration period where 84% of the P on the chromatogram was found as condensed phosphates. Essentially the same results were obtained where water extracts of each of three soils were added during equilibration, despite the fact that growth of microorganisms was noticeable in two of the three mixtures. Where solids of the same three soils were present during a 24-hour equilibration, and where the P in a portion of each solution was chromatographed, an average of 2.4% of the P^{32} on the chromatograms was associated with the condensed phosphates after equilibration where 68% of the total P on the chromatograms was found as condensed phosphates.

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One of the principal problems connected with an investigation of the hydrolysis of condensed phosphates added to soil as a fertilizer is analytical in nature. Results obtained by the usual extraction methods are of questionable validity because of incomplete extraction, hydrolysis of condensed phosphates to orthophosphate during extraction, or both. The

¹ Journal Paper No. J-4130 of the Iowa Agricultural and Home Economics Experiment Station, Ames, Iowa. Project No. 1183. Contribution from the Department of Agronomy. This work was done in cooperation with the Division of Agricultural Relations, Tennessee Valley Authority.

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technique of isotopic dilution was conceived to be a means by which the amount of orthophosphate added to soil with the condensed phosphates and produced from the condensed phosphates during subsequent hydrolysis might be measured more accurately than it could be measured with extraction methods.

One of the requirements for validity of the method of isotopic dilution is that the P present in orthophosphate must not undergo appreciable exchange with the P present in condensed phosphates. Exchange of P between these two forms has been investigated previously by Hull (2) and Quimby *et al.* (4) with results essentially negative in nature. These investigators made measurements of exchange in solutions of the phosphate salts at different degrees of acidity. If exchange in such solutions had been the only issue in the present research program, no further examination of the process would have been made. But because Van Wazer (5, p.453) noted that enzymes and colloidal gels may increase the rate of hydrolysis, and because these reactions probably are reversible to some degree, it appeared that exchange of P between orthophosphate and condensed phosphates might be enhanced by the enzymes and colloidal gels of soils.

The work reported in this paper was undertaken to verify previous findings on the exchange of P between orthophosphate and condensed phosphates in aqueous solutions and to extend the investigation to suspensions of soil. Application of the findings to development of a method for estimating the degree of hydrolysis of condensed phosphates will be described in a subsequent paper.

METHODS AND MATERIALS

Preparation of Solution Containing Condensed Phosphates

A quantity of fertilizer-grade "calcium metaphosphate" prepared by the Tennessee Valley Authority was finely ground and placed in a beaker. Water was added to cover the material to a depth of about 0.5 inch, and the beaker was covered and stored at 2 to 3°C. After a period of 15 to 50 days, a portion of the viscous material above the layer of fertilizer was removed, added to water, and filtered under pressure through a filter paper impregnated with collodion. The filtrate was diluted with water and was stored at 2 to 3°C until needed. Solutions prepared and stored in this way underwent no perceptible change in proportion of condensed phosphate and orthophosphate, and the proportion of the P present as orthophosphate amounted to 15 to 20% of the total.

Analysis for Phosphorus Present as Orthophosphate and Condensed Phosphates

The method of Dickman and Bray (1), modified in nonessential details, was used to provide a colorimetric measurement of orthophosphate in the solutions analyzed. One aliquot was analyzed without preliminary treatment to provide a value for P present in the solution as orthophosphate. To convert condensed phosphates to orthophosphate a second aliquot of the solution in question was placed in a colorimeter tube and was heated on a steam plate for 8 hours or longer with 1 ml of 6N HCl. The solution

then was neutralized with NH_4OH and analyzed for orthophosphate as indicated above. The difference between the increase in orthophosphate found in the presence of condensed phosphates and in the control was taken as condensed phosphates.

Source of Radioactivity

Radioactive P prepared by bombardment of sulfur by neutrons was obtained from the Oak Ridge National Laboratory. To ensure the presence of the P as orthophosphate, the stock solution of phosphoric acid in dilute HCl was heated on a steam plate for a period of 24 hours with an excess of bromine.

Measure of Radioactivity

Counts of P^{32} were made on dried residues obtained by evaporating a quantity of solution (usually 2 ml) in a bowl-shaped aluminum dish that had been treated with a solution of about 0.1% silicone stopcock grease in chloroform. A shielded, end-window, Geiger-Müller tube and scaler were used. Corrections for self-absorption and dead-time were neglected because of the small weights of residue and the low rates of counting. Counting was continued until 10,000 counts had been obtained or until 30 minutes had elapsed.

Paper Chromatographic Separation of Orthophosphate from Condensed Phosphates

The method of ascending paper chromatography of Karl-Kroupa (3) was used, with minor modifications, to separate the orthophosphate from condensed phosphates after contact, so that the location of the P^{32} could be determined. Modifications included (a) use of a one-dimensional run with the acid solvent only, (b) use of Schleicher and Schuell 589 Black Ribbon paper, (c) use of a spotting line 1.25 inches from the lower edge, (d) use of 10 μg of P per spot, applied in fewer than six 10- μl deliveries of solution per spot with drying of the paper between deliveries, (e) allowing the chromatographic separation to proceed at 7°C until the wet front had ascended 15.7 cm from the spotting line (7 to 8 hours), and (f) cutting the dried chromatogram into strips parallel to the spotting line and analyzing the strips for P and P^{32} . Comparable strips were composited from duplicate chromatograms (quadruplicate chromatograms in the experiment on exchange in the presence of soil) and were ashed after addition of 10 ml of 5% $\text{Mg}(\text{C}_2\text{H}_3\text{O}_2)_2$. The ash was dissolved in 1N HCl, the solution was diluted to volume in a volumetric flask, and aliquots were analyzed by the methods described above. Preliminary runs were made with reference standards, kindly supplied by E. O. Huffman, Tennessee Valley Authority, Wilson Dam, Alabama, to aid in interpretation.

RESULTS

The first experiment was conducted in the absence of soil to measure exchange of P between the orthophosphate and the various condensed phosphates present in an aqueous solution of fertilizer-grade calcium metaphosphate. A quantity of solution having a pH of about 6 and a P

concentration of about 1 g per liter was labeled with $P^{32}O_4^{---}$, shaken for 1 hour, and then allowed to stand at 25°C. Chromatograms were prepared in quadruplicate after elapse of 1, 25, and 73 hours from the addition of the P^{32} .

The results of the first experiment in Table 1 show that the radioactivity and the P were distributed differently on the chromatograms. P was concentrated near the source, where the condensed phosphates were located, and radioactivity was concentrated more distant from the source, where orthophosphate was located. Trials with reference standards showed that the condensed phosphates were located less than 6 cm from the source and that orthophosphate was located more than 6 cm from the source.

Exchange of P appeared to be negligible. The data provide two kinds of evidence. First, the radioactivity was relatively low in the portion of the chromatogram near the source, where the condensed phosphates were located. Of the total radioactivity added in the orthophosphate, only about 1% was found to be associated with the condensed phosphates. Most of the P was present as condensed phosphates, so that failure of the radioactivity to appear in these fractions did not result from limited opportunity for exchange. The radioactivity of the P in the portion of the chromatograms containing condensed phosphates amounted to 1 to 10 counts per minute per microgram compared with values of about 200 counts per minute per microgram that would have been obtained if isotopic equilibrium had been attained among all the P compounds present. Second, the number of counts per minute per microgram of P present as condensed phosphates did not increase with time of equilibration. Actually the highest number of counts was found with the shortest time available for exchange. Therefore, one may question whether the radioactivity found in the portion of the chromatograms bearing the condensed phosphates was actually present there because of incorporation into a condensed phosphate, or whether it was present as orthophosphate that had become trapped.

The second experiment involved equilibration of a solution containing condensed phosphates and orthophosphate with soil extract. Extracts of three soils were prepared by shaking 10 g of air-dry soil with 50 ml of water for 48 hours and by filtering the suspension through Whatman No. 42 paper. The soil samples were Ida silt loam (sample F-2948, pH 7.8) from Iowa, Muscatine silt loam (sample 52589, pH 4.9) from Illinois, and Waukegan silt loam (sample F-2830, pH 5.5) from Minnesota. For the equilibration, equal volumes of soil extract and solution containing condensed phosphates were mixed, and the mixture was shaken at 25°C for 0.5 hour and 72 hours before preparation of the chromatograms. The phosphate solution contained about 0.6 g of P per liter, the orthophosphate in it was labeled with P^{32} , and the reaction was about pH 6.

The results of the second experiment are shown in Tables 2, 3, 4. The values obtained are essentially the same as those obtained in the first experiment, despite the fact that after 24 hours of equilibration, growth of microorganisms was noticeable in solutions containing extracts of Muscatine and Waukegan soils. The substance in the aqueous extracts evidently caused no marked enhancement of the exchange.

The third experiment involved equilibration of a solution containing

condensed phosphates and orthophosphate directly with soil. A 5-g quantity of each of the soils mentioned above was shaken with 25 ml of water for 24 hours and then was shaken for an additional period of 24 hours after addition of 25 ml of the same tagged phosphate solution described above for the second experiment. The suspension then was centrifuged, and the supernatant liquid was passed through a collodion-impregnated filter paper under pressure. Chromatograms were prepared from the filtrate.

Results of the third experiment are shown in Tables 2, 3, and 4 alongside those of the second experiment. Of the total radioactivity found on the chromatograms, the percentages associated with the condensed phosphates were 3.1, 2.6, and 1.5 in chromatograms from the Ida, Muscatine, and Waukegan soils, respectively. These values are enough higher than the value of about 1% in the case of the pure solutions and the soil extracts that one may suspect some enhancement of exchange as a result of the presence of the soil solids during the equilibration.

DISCUSSION

The exchange of P that occurred between orthophosphate and condensed phosphates cannot be calculated from the results of the three experiments described here. One reason common to all experiments is the fact that the acid solvent used in preparing the chromatograms causes some hydrolysis of condensed phosphates to orthophosphate. The findings thus do not preclude the possibility that extensive exchange occurred between orthophosphate and one or more of the condensed phosphates that disappeared during the chromatographic separation (a small amount of hydrolysis occurred also during the period of equilibration before separation). A second reason is that in the third experiment, where the solution containing condensed phosphates and tagged orthophosphate was equilibrated directly with soil, some of the condensed phosphate was fixed by the soil, and the radioactivity of this phosphate was not measured.

The data as they stand do confirm the findings of previous investigators that the rate of exchange of P between orthophosphate and condensed phosphates is exceedingly low in vitro. Because the evidence of earlier investigators was obtained from separations that did not enhance the hydrolysis of condensed phosphates to as great an extent as did the acid solvent used in chromatography, the probability appears to be small that extensive exchange occurred between orthophosphate and one or more of the condensed phosphates but remained undetected because of hydrolysis. The method of chromatographic separation was used here because it could be used with the low concentrations of P to be employed in other research. In the proposed research, use was to be made of the method of isotopic dilution to estimate the amounts of orthophosphate added to soil in calcium metaphosphate fertilizer and produced in soil from gradual hydrolysis of the condensed phosphates in the calcium metaphosphate.

The finding of a higher proportion of the radioactivity in association with the condensed phosphates where equilibrations were made with soil than with soil extracts or water suggests that the presence of soil solids did enhance the rate of exchange. Because the condensed phosphates

Table 1. Phosphorus and radioactivity in strips of paper cut parallel with the phosphorus source from chromatograms of a solution of condensed phosphate and P^{32} -labeled orthophosphate.
Values are for the sum of two chromatograms.

Distance of strip from source in direction of movement of solution, cm	Chromatograms made 1 hour after addition of P^{32}			Chromatograms made 25 hours after addition of P^{32}			Chromatograms made 73 hours after addition of P^{32}		
	Phosphorus content, μg	Radioactivity		Phosphorus content, μg	Radioactivity		Phosphorus content, μg	Radioactivity	
		Counts per minute	Counts/min./ μg of P		Counts per minute	Counts/min./ μg of P		Counts per minute	Counts/min./ μg of P
10.5 to 13.5	17.36	23426	1349	16.59	23723	1430	17.10	24884	1455
9. to 10.5	8.46	12404	1466	8.87	11271	1271	8.56	10960	1280
7.5 to 9.	0.90	437	486	1.96	224	114	1.33	240	180
6. to 7.5	1.16	126	109	1.50	56	37	1.52	42	28
4.5 to 6.	10.46	100	10	10.54	44	4	11.54	44	4
3. to 4.5	8.70	76	9	8.93	21	2	9.16	46	5
1.5 to 3.	31.98	62	2	21.53	36	1	35.02	52	2
0. to 1.5	77.19	111	1	73.51	38	1	72.86	116	2
-1.5 to 0.	23.06	52	2	29.70	82	3	19.66	33	2

Table 2. Phosphorus and radioactivity in strips of paper cut parallel with the phosphorus source from chromatograms of solutions of condensed phosphate and P^{32} -labeled orthophosphate after equilibration with Ida silt loam.

Distance of strip from source in direction of movement of solution, cm	Chromatograms of solution after equilibration with a water extract of soil (sum of 2 chromatograms)					Chromatograms of solution after equilibration with soil for 24 hours (sum of 4 chromatograms)				
	Chromatograms made 0.5 hour after addition of P^{32}			Radioactivity		Chromatograms made 72 hours after addition of P^{32}			Radioactivity	
	Phosphorus content, μg	Counts per minute	Counts/min. μg of P	Phosphorus content, μg	Counts per minute	Counts/min. μg of P	Phosphorus content, μg	Counts per minute	Counts/min. μg of P	
10.5 to 13.5	15.88	36398	2292	21.30	36463	1712	30.03	46363	1544	
9. to 10.5	7.86	17068	2172	9.77	17866	1829	5.02	7252	1445	
7.5 to 9.	1.52	334	220	1.50	263	175	1.84	445	242	
6. to 7.5	1.08	33	31	1.28	86	67	2.94	344	117	
4.5 to 6.	10.14	94	9	12.04	117	10	6.87	445	65	
3. to 4.5	8.56	8	<1	9.11	20	2	17.17	297	17	
1.5 to 3.	31.08	7	<1	36.79	-5	<1	25.52	371	14	
0. to 1.5	51.43	110	2	67.32	134	2	23.07	393	17	
-1.5 to 0	19.66	60	3	16.38	20	1	12.86	235	18	

Table 3. Phosphorus and radioactivity in strips of paper cut parallel with the phosphorus source from chromatograms of solutions of condensed phosphate and P^{32} -labeled orthophosphate after equilibration with Muscatine silt loam.

Distance of strip from source in direction of movement of solution, cm	Chromatograms of solution after equilibration with a water extract of soil (sum of 2 chromatograms)				Chromatograms of solution after equilibration with soil for 24 hours (sum of 4 chromatograms)			
	Chromatograms made 0.5 hour after addition of P^{32}		Chromatograms made 72 hours after addition of P^{32}		Chromatograms made 72 hours after addition of P^{32}		Chromatograms made 72 hours after addition of P^{32}	
	Phosphorus content, μg	Radioactivity Counts per minute μg of P	Phosphorus content, μg	Radioactivity Counts per minute μg of P	Phosphorus content, μg	Radioactivity Counts per minute μg of P	Phosphorus content, μg	Radioactivity Counts per minute μg of P
10.5 to 13.5	14.29	27941	1955	28858	18.96	35943	1896	
9. to 10.5	10.71	19634	1833	16820	18.96	37699	1988	
7.5 to 9.	1.29	434	336	446	2.44	2123	870	
6. to 7.5	1.13	98	87	81	3.88	554	143	
4.5 to 6.	8.14	83	10	92	7.50	281	38	
3. to 4.5	9.96	31	3	70	21.03	196	9	
1.5 to 3.	30.14	12	<1	89	24.32	274	11	
0. to 1.5	68.52	82	1	155	35.18	672	19	
-1.5 to 0	31.88	70	2	138	26.47	595	22	

Table 4. Phosphorus and radioactivity in strips of paper cut parallel with the phosphorus source from chromatograms of solutions of condensed phosphate and P^{32} -labeled orthophosphate after equilibration with Waukegan silt loam.

Distance of strip in direction of movement of solution, cm	Chromatograms of solution after equilibration with a water extract of soil (sum of 2 chromatograms)						Chromatograms of solution after equilibration with soil for 24 hours (sum of 4 chromatograms)			
	Chromatograms made 0.5 hour after addition of P^{32}			Chromatograms made 72 hours after addition of P^{32}						
	Radioactivity			Radioactivity			Radioactivity			
	Phosphorus content, μg	Counts per minute	Counts/ min./ μg of P	Phosphorus content, μg	Counts per minute	Counts/ min./ μg of P	Phosphorus content, μg	Counts per minute	Counts/ min./ μg of P	
10.5 to 13.5	16.60	34644	2087	21.23	43640	2056	22.61	23435		1036
9. to 10.5	7.95	16621	2091	5.24	9016	1721	29.64	30272		1021
7.5 to 9.	1.44	218	151	1.36	227	167	1.57	1211		771
6. to 7.5	1.32	58	44	1.77	58	33	2.58	220		85
4.5 to 6.	10.30	65	6	11.14	60	5	6.63	90		14
3. to 4.5	8.56	12	1	10.16	12	1	16.90	139		8
1.5 to 3.	30.44	46	1	34.60	35	1	28.28	98		4
0. to 1.5	71.70	106	1	70.89	188	3	20.16	273		14
-1.5 to 0	24.70	45	2	25.11	62	2	20.88	232		11

were retained to a considerable extent by the soil solids, and because the radioactivity of this portion is unknown, it is possible that unequal distribution of radioactivity between the solid and solution portions was responsible, at least in part, for the apparent enhancement of exchange.

For the purpose of the authors, interest in the appearance of P^{32} in the portion of the chromatograms containing the condensed phosphates is connected principally with the error in isotopic-dilution estimates of orthophosphate that would be caused if the P^{32} were actually incorporated into the condensed phosphates. Again no exact quantitative interpretations can be made from the data, but a general idea of the situation can be derived from the following considerations. In the method of isotopic dilution, the quantity, P_{solid} , of orthophosphate in the soil solids that has equilibrated with tagged orthophosphate in the solution may be estimated by the equation

$$(P_{\text{solid}}) = (P_{\text{solution}})(a_0 - a_{\text{final}}) / (a_{\text{final}})$$

where P_{solution} is the orthophosphate found in solution at the end of the equilibration period, a_0 is the specific activity of the orthophosphate in solution at the end of the equilibration period if no loss of P^{32} from the orthophosphate in solution had occurred during the equilibration period, and a_{final} is the specific activity of the orthophosphate in solution at the end of the equilibration period. In the application of the method of isotopic dilution made by the authors, no chromatographic separation is carried out; instead, an aliquot of the solution is counted directly. The orthophosphate in solution is measured independently of the condensed phosphates that may be present. Values of P_{solution} and a_0 thus are measured with only the usual errors, but values of a_{final} are subject to two biases, namely, loss of P^{32} from orthophosphate into condensed phosphates and inclusion of the radioactivity of the solution-phase condensed phosphates with that of the solution-phase orthophosphate.

These considerations make possible an estimate of the error introduced into measurements of orthophosphate by isotopic dilution. Calculations of error may be made for various proportions of orthophosphate and condensed phosphate and for different proportions of each form in the solution. In the experimental work of the authors (to be reported separately), the apparent distribution of 100 parts of P added to different soils as calcium metaphosphate fertilizer and found after a period of incubation was 15 to 39 as orthophosphate in solution, 13 to 76 as orthophosphate in the solids, 0 to 15 as condensed phosphates in solution, and -8 to 54 as condensed phosphates in the solids. Calculations indicated that under these conditions the error in the estimates of orthophosphate associated with exchange amounted to less than 6%, a value that was not considered excessive.

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PUBLICATIONS OF MEMBERS OF THE STAFF
OF THE IOWA STATE UNIVERSITY FOR
THE ACADEMIC YEAR 1960-61

Certain summaries and indices are of interest in a survey of the publications of members of the staff of an educational and research institution such as the Iowa State University. The publications are listed in alphabetic order under the names of the senior authors. Junior authors are also listed alphabetically with cross reference to senior author.

SUMMARY

Number of individuals listed	775
Number of publications.	810
Number of publications with single author	337
Number of publications with joint authorship	473
Number of departments or fields represented in publications	49
Number of individuals who serve as editors or on the editorial staff of one or more scientific or technical periodicals	56

Individuals thus serving are: S. Aronoff; R. E. Atkins; J. C. Ayres, E. R. Baumann; C. A. Black, R. E. Buchanan; Lee Burchinal; O. N. Carlson; A. H. Daane; Paul A. Dahm; G. C. Danielson; Harvy Diehl; Ercel Eppright; V. A. Fassel; Richard H. Forsythe; Henry Gilman; Carol A. Greiner; R. L. Handy; Arnold O. Haugen; Virgil W. Hays; John Heer; Clayton J. Herman; Paul J. Hermann; M. S. Hofstad; N. L. Jacobson; Will Jumper; Oscar Kempthorne; William F. Kenkel; Harry H. Knight; Oliver A. Knott; Lee Kolmer; Francis A. Kutish; George W. Ladd; W. E. Larson; Wilbur L. Layton; W. R. Lockhart; Mary Lyle; John P. Mahlstede; Dale McCay; R. M. Melampy; A. W. Nordskog; Don C. Norton; J. B. Page; John T. Pesek, Jr.; Richard Phillips; Walter C. Rothenbuhler; Marr D. Simons; Frederick G. Smith; David W. Staniforth; Melvin J. Swenson; John F. Timmons; Gerhard Tintner; C. R. Weber; Milton W. Weller; Richard J. Zbaracki; C. H. Werkman.

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